DEVELOPMENT OF MICROWAVE-ASSISTED TRANSITION METAL FERRITES ($NiMFe_2O_4$; M = Mn, Co, Cu, Zn) FOR ENERGY STORAGE APPLICATION

A SYNOPSIS

Submitted by

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(Reg. No. 9202026003)

in partial fulfilment for the award of the degree

of

DOCTOR OF PHILOSOPHY

DEPARTMENT OF PHYSICS

SCHOOL OF ADVANCED SCIENCES

KALASALINGAM ACADEMY OF RESEARCH AND EDUCATION

(DEEMED TO BE UNIVERSITY)

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TAMILNADU, INDIA

JUNE 2024

1. INTRODUCTION

Over the last decades, there is a serious necessity for clean, environmentally an effective long-lasting energy sources due to the impact of non-renewable energy resources in environmental pollution, the exhaustion of non-renewable energy resources, and the continuous demand for oil [1–4]. In the future, the electronic systems with the inevitability of energy storage devices and must be improved by emerging novel materials and evolving of nanoscale electrochemical interfaces. One of the fascinating and significant ways of producing electrical energy is by electrochemical reactions and it is stored in the fuel cells, supercapacitors, or batteries in the portable electronic devices in hybrid vehicles [5–6]. The deeper fundamental knowledge and new phases of investigation in the energy materials are essential to fabricate supercapacitors with high-performance capabilities [7,8].

Because of this, recent researches in energy storage devices have been promoted by great interests in academics and industry for the characteristics like long lifetime, high energy capacities, and high-rate capability. Lithium-ion batteries, supercapacitors (SCs), fuel cells, and solar cells are some of the most attractive devices currently under development. Furthermore, the development of state-of-the-art energy storage devices is becoming more and more important and many researchers have focused on the use of advanced energy storage devices.

` Nowadays, the SCs has attracted as a gift in energy storage devices due to many advanced characters including fast charge/discharge rate, high power density, long cycle stability, and eco-friendly [9]. SCs by its energy-storing behavior can be categorized as electrical double-layer capacitor (EDLC) and the pseudocapacitor. In EDLCs, the charge storage is by ion adsorption/desorption at the electrode/electrolyte interface and consists of carbon electrodes. The charge is stored by redox reactions at the electrode/electrolyte interface in the case of pseudocapacitors and employs transition metal oxides or conducting polymers as electrode materials [10,11].

The performance of SCs is significantly affected by many factors, such as the electrochemical properties of the electrode materials, the choice of electrolyte, and the potential window of the electrodes. Large research efforts have been directed to facilitate effective electron transport and ion diffusion with structural designs suitable for the development of advanced materials for SC electrodes [12].

Researchers have been taken considerable efforts to scheming efficient electrochemical storage devices to enhance the capacitance by optimizing several determining sources like stimulating quicker electron movement through porous channels, increasing surface area, reducing agglomeration of nanomaterials and interfacial resistance, etc. [13].

Extensive research has shown the significance of transition metal oxides as efficient supercapacitor electrode materials due to different factors like cheap, eco-friendly, good structural, electronic and mechanical properties. Due to these reasons, metal oxides like NiO, ZnO, Co_3O_4, Fe_2O_3 have been studied extensively. The ions and electrons present in electrode materials are involved in the fast and reversible faradaic reaction during charge storage [14].

Though these transition metal oxides are cheaper, but the low specific capacitance and low electrical conductivity demand further improvements. Therefore, this research work deals with the synthesis of transition metal ferrites nanomaterials as electrodes for Supercapacitor. Compared to bulk materials, nanostructured materials as an electrode in supercapacitor will exhibit superior kinetics and activity because of their higher specific surface area and smallest ion and electron transport pathway. In particular, the electrode material with high specific capacitance and wide range of operating potential are desirable to fabricate a supercapacitor. Ferrites with mixed oxidation states have proved as promising electrodes in supercapacitors. The ferrite materials are also being considered as potential electrodes in supercapacitor because of their different oxidation states, low price, environmental benignity, and their large abundance.

2. LITERATURE REVIEW

The research for high-performance super capacitive materials that can deliver as much energy density as conventional capacitors and conventional batteries has never landed among the research community. Among various electrode materials, transition metal chalcogenides have attracted significant attention due to their intricate electrochemical properties such as rich redox chemistry, high valence levels, high electronic conductivity compared to their oxide counterparts, fast ion diffraction kinetics and morphological diversity; However, they have rate performance, cyclability, surface area, and agglomeration issues. Thirumurugan Arun et al., 2021 [15], The cyclic voltammogram, galvanostatic charge/discharge and electrochemical impedance measurements were carried out to evaluate the electrochemical characteristics of prepared $NiFe_2O_4$ nanoparticles for supercapacitor application. The highest specific capacitance of 277 F/g was observed for the $NiFe_2O_4$ nanoparticles that prepared with 50 % of ferric ions (NF50). From the cyclic stability study, 101 % of capacitance retention was demonstrated up to 5000 cycles. An asymmetric two-electrode cell that fabricated with NF50 exhibited a maximum specific capacitance of 56 F/g (with an applied current density of 1 A/g) and higher energy density of 22.5 Wh/ Kg (with a power density of 0.85 kW/Kg). The cyclic stability study of the cell revealed the increased capacity retention (126 % up to 5000 cycles) due to the particle size reduction.

Gao et al., 2018 [16] $NiFe_2O_4$ (NFO) with different morphologies were successfully synthesized via a facile hydrothermal method of different precipitants. The specific capacitance of NFO-U could arrive 240.9 F/g at the current density of 1 A/g. It was worth noting that the cycle performance of NFO-U was excellent, of which the specific capacitance improved to 128 % after 2000 cycles. In addition, an asymmetric supercapacitor (ASC) system was constructed utilizing NFO-U as positive electrode while activated carbon (AC) as negative electrode. The two-electrode system showed a good performance with energy density of 10.15 Wh/kg at a power density of 140 W/kg. Therefore, the construction of sheet-like structures greatly improved the electrochemical

performance of $NiFe_2O_4$ materials. Ghasemia et al., 2018 [17] Electrochemical properties of the prepared nanospheres were studied in order to realize their suitability and susceptibility as an electrode material for supercapacitor applications. $NiFe_2O_4$ nanospheres showed a high specific capacitance of 122 F/g and a great specific energy of 16.9 Wh/kg at a high current density of 8.0 A/g. The maximum specific capacity of the nanospheres was even more, which can reach 137.2 F/g at 4 A/g of current density. The results showed an excellent long-term cycling stability for the $NiFe_2O_4$ nanospheres-based electrodes. The capacitance did not decrease, compared to the initial value during 100 galvanostatic charge–discharge cycles. The electrochemical performances were measured using three electrode configurations by cyclic voltammetry (CV) measures in 6M KOH electrolyte. CV tests have been performed for three different loading masses of the obtained $CoFe_2O_4$ as active material on a Ni foam current collector. These ferrites nanoparticles show high values of specific capacitance, the best result being obtained for the lowest loading mass, with 315 F/g.

Shrikant et al., 2016 [18]. $ZnFe_2O_4$ thin film electrode material for supercapacitor through electrochemical analysis. $ZnFe_2O_4$ thin film exhibited a specific capacitance of 471 Fg^{-1} at a scan rate of 5 mVs^{-1} in 1 M NaOH aqueous solution. Further, $ZnFe_2O_4$ solid-state symmetric (SSS) supercapacitor device demonstrated voltage window of 1.0 V with specific capacitance of 32 Fg^{-1}, energy density of 4.47Wh/kg and power density of 277W/kg at 1 Ag^{-1} current density. Nawaz et al., 2021 [19]. prepared $Ni_{0.5}Zn_{0.5}Fe_2O_4$ is synthesized as binder-based (NZF) and binder-free electrodes (NZF@NF). The binder-free electrode is directly synthesized on nickel foam via facile hydrothermal techniques. The NZF@NF electrode has outperformed the binder-based NZF electrode in terms of electrochemical performance owing to the 3D interconnected structure of the nickel foam. The NZF@NF electrode has delivered a high specific capacity of 504 F g^{-1} at the current density of 1 A g^{-1}, while its counterpart has delivered a specific capacity of 151 F g^{-1} at the same current density. Bhujun et al., 2016 [20] Nano crystallites of three mixed ternary

transition metal ferrites (MTTMF) were prepared by a facile sol–gel method and adopted as electrode material for supercapacitors. The electrochemical performance of the nanomaterials was evaluated using a two-electrode configuration by cyclic voltammetry, electrochemical impedance spectroscopy and galvanostatic technique in 1 M KOH electrolyte and was found to be in the order of: $CuCoFe_2O_4$ > $NiCoFe_2O_4$> $NiCuFe_2O_4$. A maximum specific capacitance of 221 Fg^{-1} was obtained with $CuCoFe_2O_4$ at a scan rate of 5 mV s^{-1}. In addition to an excellent cycling stability, an energy density of 7.9 kW kg^{-1} was obtained at a current density of 1 Ag^{-1}. The high electrochemical performance of the MTTMF nanocomposites obtained indicates that these materials are promising electrodes for supercapacitors.

3. RESEARCH PROBLEM

High-efficiency electrochemical device is one of the most important needs of today, and the role of electrodes is massive in its development. Therefore, the properties of these electrodes depend on the properties of the materials from which they are made. Especially the surface-to- volume ratio and structure properties make it very suitable for redox mechanism (diffusion of ions/electrons, adsorption/desorption). So, both these properties are easily possible in nanoparticles. Transition metal ferrites for supercapacitor applications using microwave combustion method is a fascinating area of study. In this method, transition metal ferrites nanoparticles are synthesized through a combustion process using microwave irradiation. This technique offers several advantages, including rapid synthesis, high purity of the resulting materials, and control over the particle size and morphology. One key research problem in this area is optimizing the microwave combustion process to achieve transition metal ferrites materials which improved specific capacitance, excellent rate capability, and long-term cycling stability for supercapacitor application. Scientist are investigating the influence of different synthesis parameters on the microstructure, surface area, and electrochemical behavior of the ferrite materials to maximize their energy storage capacity and efficiency.

Overall, research on transition metal ferrites for supercapacitors via microwave combustion method aims to develop cost-effective, high-performance electrode materials that can meet the increasing demand for energy storage devices with rapid charging capabilities and long cycle life.

4. OBJECTIVE OF THE RESEARCH WORK

In the next few years, our fossil fuel powered vehicles and home heating will have to switch to electric power. But it also suffers from a major drawback. The idea is to store this electrical energy and make it available for long-term use which is effectively done by Supercapacitor. However, to increase its efficiency and meet the demand for clean energy, its main component electrodes are produced with the help of biomass, which gives more electrochemically active sites. Therefore, there is a need for nanoparticles that can be produced by simple, inexpensive, and environmentally friendly microwave combustion synthesis. All these reasons motivate us to develop transition metal ferrites nanoparticles as an electrode for Supercapacitor application. Therefore, the following processes have been carried out in this research.

- To synthesize transition metal ferrites ($NiMFe_2O_4$) (M = Mn, Cu, Zn, Co.) using the microwave-assisted combustion method
- To study the structural properties of synthesized $NiMFe_2O_4$ using XRD and FTIR analyses
- To characterize the synthesized $NiMFe_2O_4$ using various techniques such as scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDX), high-resolution transmission electron microscopy (HRTEM), and X-ray photoelectron spectral (XPS) analyses
- To evaluate the electrochemical performance of $NiMFe_2O_4$ as supercapacitor electrodes, including specific capacitance, cyclic stability, rate capability, and charge-discharge efficiency.

5. Organization of the thesis

The thesis can be summarized in eight chapters and is explained below.

Chapter 1 – Introduction

Chapter 1 discuss the background of the transition metal ferrites nanoparticles as an alternative source for energy storage applications due to the demand for fossil fuels. Concerning this, the importance and the focus of this research work on transition metal ferrites nanoparticles are discussed. Due to the environmentally friendly nature and cost- effectiveness of NPs management possible in the microwave combustion method, hence transition metal ferrites nanoparticles and general discussion about their properties are discussed. The detailed description of energy storage devices such as a capacitor, Supercapacitor, and their classification namely Electric Double Layer Capacitor (EDLC), Pseduocapacitor, hybrid Supercapacitor, and assembling of Supercapacitor is deliberate and its general properties and merits, demerits are summarized.

Chapter 2 – Literature review

Chapter 2 discuss the literature review of research work entitled "Development of microwave assisted synthesis of transition metal ferrites nanoparticles for energy storage applications" In this chapter, discussed the outline of the literature review. The literature study has focused on fabricate transition metal ferrites with optimized properties for supercapacitors. The microwave combustion method has been extensively studied to tailor the morphology, surface area, and crystal structure of the ferrites nanoparticles to enhance their electrochemical performance

Chapter 3 – Materials and Methods

In this Chapter 3, materials are used to carry out the research and the ternary transition metal ferrites are prepared by microwave combustion technique are briefly discussed in Figure 1. The basic, advanced characterization techniques of nanoparticles such as measurement, XRD, FTIR, SEM, HR-TEM, XPS, and VSM analysis are discussed with a schematic diagram. Inaddition, the fabrication of active electrodes using synthesized NPs by doctor blade technique and working of an electrochemical workstation with instrumental setup, its characterization techniques are briefly discussed in Figure 2. The detailed characterization results are used to understand the functional group, morphological, structural, and magnetic properties of prepared materials. The studied results are more suitable for Electrochemical performance.

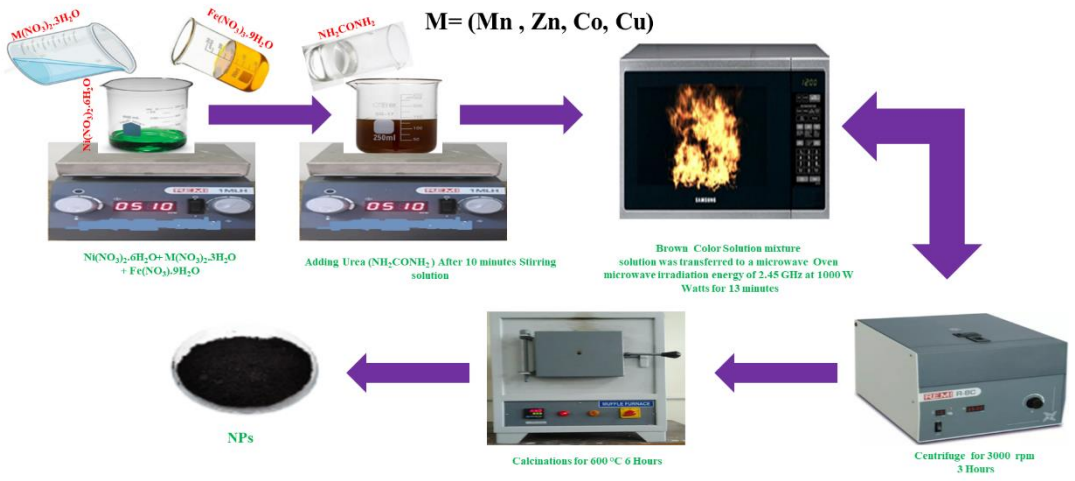
M= (Mn , Zn, Co, Cu)
Ni(NO3)2.6H2O+ M(NO3)2.3H2O + Fe(NO3).9H2O
Adding Urea (NH2CONH2) After 10 minutes Stirring solution
Brown Color Solution mixture solution was transferred to a microwave Oven microwave irradiation energy of 2.45 GHz at 1000 W Watts for 13 minutes
Centrifuge for 3000 rpm 3 Hours
Calcinations for 600 °C 6 Hours
NPs

Figure 1 Microwave combustion technique

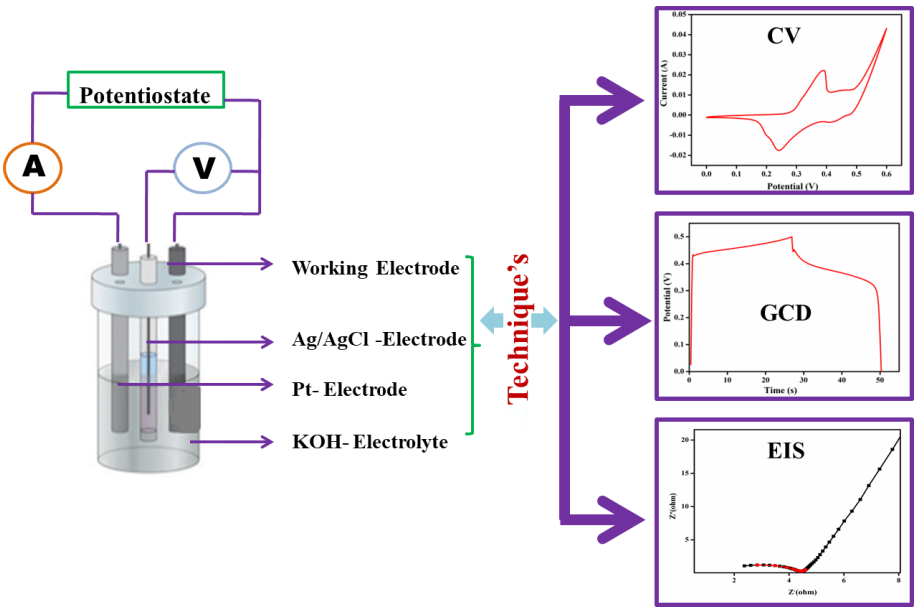

Figure 2 Electrochemical characterization

Chapter 4 – Investigations on transition metal ferrite ($NiCoFe_2O_4$) as potential electrode for energy storage application

Chapter 4 deals with nanostructured ternary transition metal ferrite ($NiCoFe_2O_4$) nanomaterial were synthesized by simple Microwave assisted irradiation method. The structural phase identification of the sample was confirmed with XRD pattern. Nanocrystalline nature of $NiCoFe_2O_4$ was confirmed using Scherrer formula using PXRD peaks. Particle size of the material is found to be 37 nm. The observed peaks are matched with the earlier reports of $NiCoFe_2O_4$. Structural bond formation was identified through FTIR. The SEM images clearly depicted nanosphere like morphology. HRTEM micrograph reveals that the particles are nano sized and it is in hexagonal shape morphology. The VSM study confirmed the prepared material as ferromagnetic. From the electrochemical study, Cyclic voltammetry curve of $NiCoFe_2O_4$ given in Figure 3. The highest Csp value obtained as 110.2 F/g at the current density of 1 A/g. From charge discharge graph, the maximum specific capacitance value calculated was 110.2 F/g at 1 A/g. The $NiCoFe_2O_4$//AC ASC had extremely excellent cycling stability of 94%

retention even after 5000 cycles. The modified $NiCoFe_2O_4$ electrode showed excellent electrochemical performance, which presents to be a great potentials and values for practical applications on supercapacitors.

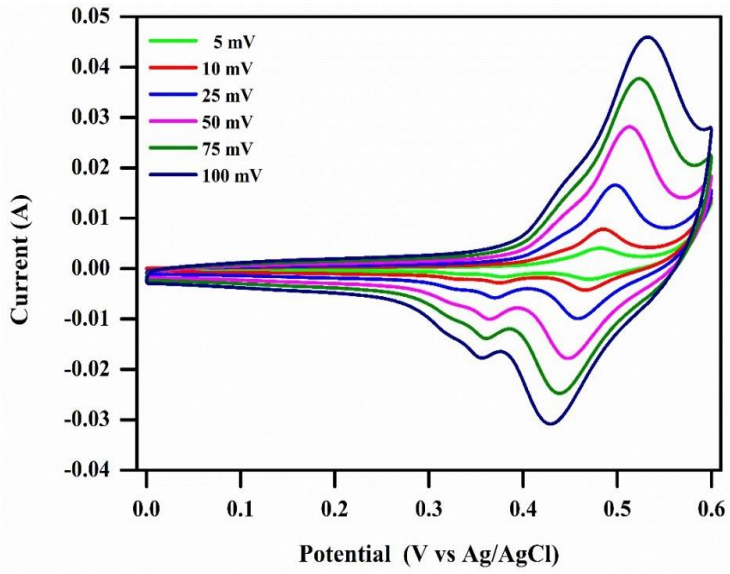

Figure 3 Cyclic voltammetry curve of $NiCoFe_2O_4$

Chapter 5 –Facile synthesis of $NiMnFe_2O_4$ for supercapacitor application

Chapter 5 describes the Nanosized ternary transition metal ferrite ($NiMnFe_2O_4$) has been synthesized using an easy and inexpensive microwave-assisted combustion method. The powder XRD confirms the nanoparticles as in spinal cubic structure with space group $Fd\bar{3}m$. FTIR has analyzed to understand the vibration of metal ions in the prepared $NiMnFe_2O_4$. The elemental composition and surface morphology of the nanoparticles have been studied by scanning electron microscopy (SEM) equipped with an energy-dispersive X-ray (EDX) analysis. The HR-TEM showed the $NiMnFe_2O_4$ particle in a hexagonal shape and is observed as a dark field mode corresponding to $NiMnFe_2O_4$.The electrochemical properties of the prepared metal ferrite have been studied by electrochemical impedance spectroscopy (EIS), Galvanostatic charge-discharge (GCD) and cyclic voltammetry (CV).

The electrochemical impedance spectroscopy of $NiMnFe_2O_4$ given in Figure 4. The specific capacitance of the modified electrode is found to exhibit the highest value of 476 F/g at 5 mV/s.

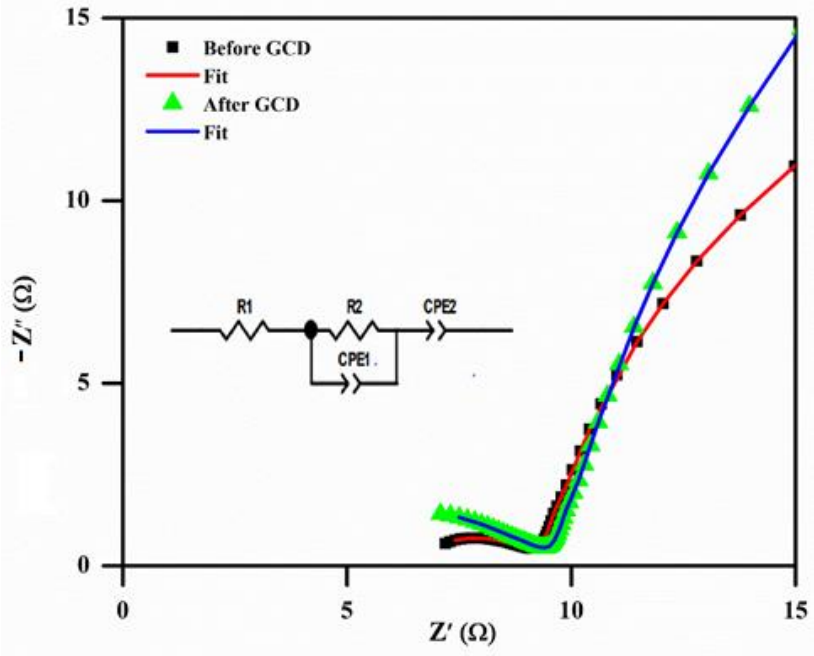

Figure 4 Electrochemical impedance spectroscopy of $NiMnFe_2O_4$

Chapter 6 – Microwave combustion synthesis of NiZnFe2O4 for Supercapacitor application

Chapter 6 describes the magnetic and electrochemical properties of $NiZnFe_2O_4$ nanoparticles (NPs) were synthesized by the microwave combustion method. The structural and morphological studies of $NiZnFe_2O_4$ NPs were characterized by X-ray diffraction (XRD) and scanning electron microscope (SEM). The synthesized NPs were crystallized in the spinel structure with the $Fd\bar{3}m$ space group. The magnetic properties were examined by a vibrating sample magnetometer (VSM). The magnetic data analysis confirms that the coercivity of the synthesized NPs is nearly zero at room temperature, the saturation magnetization values (Ms) 87.37. Electrochemical studies reveal that $NiZnFe_2O_4$ NPs have superior electrochemical performance. The asymmetric supercapacitor device was fabricated using the prepared sample and active carbon. The $NiZnFe_2O_4$//AC carried a high specific

capacitance of 73.28 F/g at a scan rate of 5 mV/s. From the Galvanostatic charge–discharge analysis in Figure 5, the $NiZnFe_2O_4$//AC provided a specific capacitance of about 32.91 F/g at 6A/g with an energy density of 3.56 Wh/kg and power density of 2051.28 W/kg.

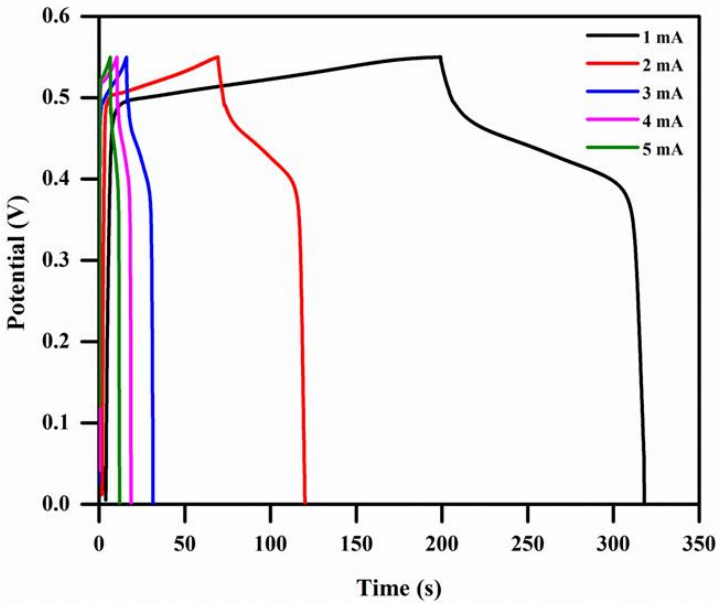

Figure 5 Galvanostatic charge discharge curve of $NiZnFe_2O_4$

Chapter 7 – microwave assisted synthesis of $NiCuFe_2O_4$ for Energy Storage Applications

Chapter 7 deals with the $NiCuFe_2O_4$ nanoparticles (NPs) have been prepared by the microwave combustion method and used as an electrode material for supercapacitor applications. The formation of a spinel cubic ferrite with an agglomeration of particle-like morphological structures has been affirmed by the PXRD and SEM analyses. The presence of functional groups specific to the nickel-copper ferrite formed during the microwave combustion process has been investigated by the FTIR. HR-TEM revealed hexagonal shaped aggregated particles. The magnetic behavior of the spinal $NiCuFe_2O_4$ NPs revealed soft ferromagnetism. The chemical valence state of each element in $NiCuFe_2O_4$ nanoparticles were determined using X-ray photoelectron spectroscopy (XPS). In a three-electrode configuration, using a 3 M KOH

electrolyte, the $NiCuFe_2O_4$ electrode demonstrated a high specific capacitance of 220.6 F/g at a scan rate of 5 mV/s with excellent cycling stability. The two-electrode configuration the device demonstrated a maximum power density of 2419 W/kg and a maximum energy density of 8.78 Wh/kg. These notable results demonstrate the high electrochemical characteristics of nickel-copper ferrite as a pioneering spinel cubic ferrite electrode as a potential supercapacitor.

Chapter 8 – Summary and Conclusion

This chapter deals about the overall summary and conclusion of this thesis work. Nano crystallites of transition metal ferrites were prepared by a microwave assisted combustion method and adopted as electrode material for supercapacitors. The microwave irradiation technique is a currently familiar, quick and simple technique used to prepare various inorganic compounds. X-ray diffraction (XRD) analysis revealed the formation of a single-phase spinel ferrite in $NiMnFe_2O_4$, $NiCoFe_2O_4$, $NiCuFe_2O_4$ and $NiZnFe_2O_4$ with the $Fd\bar{3}m$ space group. The phase development of the samples was determined using Fourier transform infrared (FT-IR). The surface characteristics and elemental composition of the nanoparticles have been studied by means of scanning electron microscopy (SEM), as well as energy dispersive spectroscopy (EDS) and high-resolution transmittance electron microscopy (HR-TEM). The magnetic properties of all the prepared spinel nanostructured ferrites have been evaluated by VSM analysis. The electrochemical performance of the nanomaterials was evaluated using a three-electrode and two electrode configuration system. These investigations are aimed at leveraging the desirable properties of these ferrites to enhance the performance of the supercapacitors. A maximum specific capacitance of 476 F/g was obtained with $NiMnFe_2O_4$ at a scan rate of 5 mV/s. In addition to an excellent cycling stability. The high electrochemical performance of the ternary transition metal ferrites obtained indicates that these materials are promising electrodes for supercapacitors.

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LIST OF PUBLICATIONS

1. **M. Mayakkannan,** A. Murugan, A. Shameem, V. Siva, S. Sasikumar, S. Thangarasu, S. Asath Bahadur, “Investigations on ternary transition metal ferrite: $NiCoFe_2O_4$ as potential electrode for supercapacitor prepared by microwave irradiation method”, Journal of Energy Storage 2021, 44, 103257.
2. **M. Mayakkannan,** V. Siva, A. Murugan, A. Shameem, S. Thangarasu, S. Asath Bahadur, “Microwave-assisted synthesis of ternary transition metal ferrite: Structural, morphological, optical, magnetic and electrochemical properties”, Physica E: Low-dimensional Systems and Nanostructures 2023, 147, 115573.
3. **M. Mayakkannan,** V. Siva, A. Murugan, A. Shameem, M. Venkateshwarlu, S. Asath Bahadur and S. Thangarasu, “One step microwave combustion synthesized spinel nano NiZnFe2O4 as an electrode material for supercapacitors with superior performance”, J Mater Sci: Mater Electron 2023, 34, 1397.
4. **M. Mayakkannan,** S. Thangarasu, V. Siva, A. Murugan, A. Shameem, and S. Asath Bahadur, “Facile microwave combustion synthesis of nanocrystalline Ni-Cu spinel ferrite as an asymmetric supercapacitive device”, Materials Science & Engineering B 306 (2024) 117448.